High-Temperature Natural Antioxidant Improves Soy Oil for Frying

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ABSTRACT: The objectives of this study were to determine the frying stability of soybean oil (SBO) treated with a natural citric acid-based antioxidant, EPT-OILShield^{TM} able to withstand high temperatures and to establish the oxidative stability of food fried in the treated oil. Soybean oil with 0.05% and 0.5% EPT-OILShield and an untreated control SBO were used for intermittent batch frying of tortilla chips at 180 °C for up to 65 h. Oil frying stability was measured by free fatty acids (FFA) and total polar compounds (TPC). Chips were aged for up to 4 mo at 25 °C and evaluated for rancid flavor by a 15-member, trained, experienced analytical sensory panel and for hexanal content as an indicator of oxidation. Oil with 0.05% EPT-OILShield had significantly less FFA and TPC than the control. The effect of EPT-OILShield was apparently retained in aged chips because hexanal levels were significantly lower in chips fried in oil with 0.05% EPT-OILShield than in chips fried in the control. Tortilla chips fried in the control were rancid after 2 mo at 25 °C at sampling times evaluated from 25 to 65 h; however, chips fried in oil with 0.05% EPT-OILShield and used for 65 h were described as only slightly rancid after 4 mo. Gamma tocopherol levels were significantly higher in the chips fried in the oil with 0.05% EPT-OILShield than in the control, helping to inhibit oxidation in the tortilla chips during storage.

Keywords: antioxidant, frying, oxidation, shelf life, soybean oil

Introduction

Concern about *trans* fatty acids is causing food manufacturers and restaurants to seek alternative frying oils that have good oxidative stability without the need for hydrogenation. Options to enhance frying oil stability include oils with modified fatty acid compositions such as high oleic and mid-oleic sunflower, high oleic/low linolenic canola, high oleic safflower, and low linolenic soybean, and mid-oleic/ultra low linolenic soybean. Although these oils show improved frying stability compared to the unmodified original oils, they usually are higher in cost and limited in availability now. To avoid hydrogenated fats containing *trans* fatty acids and the high costs of alternatives, some frying operations are opting for oxidatively unstable soybean and canola oils that have high amounts of polyunsaturated fatty acids. However, problems such as poor flavor of fried food and potential unhealthful degradation products can occur with these unstable oils. In fact, one frying study reported that an oil should not be considered suitable for frying if it contains more than 2% linolenic acid (Sebedio and others 1990). Additional options are needed to improve these unstable oils for frying. Oil additives are a potential alternative for inhibiting frying oil oxidation. For example, antioxidants such as butylated hydroxyanisole (BHA), butylated hydroxytoluene (BHT), and tertiary butylhydroquinone (TBHQ) can protect oils at ambient temperatures and are used in frying oils. Augustin and Berry (1983) reported that results of chemical and instrumental tests showed few differences between untreated palm olein and the same oil treated with 200 ppm or less of BHT and BHA. However, they found that palm olein lost 70% of the original BHT and 60% of BHA after 8 h of heating. Other researchers have also reported that BHA, BHT, and TBHQ volatilized during frying and were ineffective in enhancing oxidative stability of frying oil (Hawrysh and others 1990). Freeman and others (1973) statically heated sunflower oils containing 200 ppm of BHT or 1 ppm methyl silicone. They reported no protective effect from the antioxidant, whereas silicone had a significant protective effect in inhibiting oxidation.

Natural antioxidants have been used in frying oils (Jaswir and others 2000) in palm olein. These researchers showed that a combination of rosemary, sage, and citric acid was the most effective in retarding degradation of fatty acids, but citric acid alone was only slightly better than no additives. Warner and others (1985) also reported that THBQ as well as citric acid used during heating of soybean oil had no effect compared to soybean oil with no additives. The lack of effect of citric acid during frying is not unexpected because citric acid also volatilizes at the high temperatures as do the chemical antioxidants. Citric acid is added during processing to edible oils at levels of 0.005% to 0.01% to act as a metal chelator and thereby helps protect the oil from oxidation (Brekke 1980). However, citric acid decomposes at temperatures above 150 °C, so it needs to be added to deodorized oil at 130 °C or less during cooling after deodorization (Frankel 2005). In addition to citric acid's role as a metal chelator, Jaswir and Che Man (1999) reported that citric acid, together with an antioxidant, could be considered as a synergist for enhancing antioxidant activity in palm oil. They also found that citric acid alone in oil used for heating and frying was not effective. However, Cerretani and others (2008) conducted a study using SBO, containing the same citric acid-based antioxidant evaluated in this study (at 0.05% and 0.1%), to fry French fried potatoes at 190 °C. Based on chemical analyses of the oils, they reported that the antioxidant was heat resistant to 190 °C and that both primary

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Names are necessary to report factually on available data; however, the USDA neither guarantees nor warrants the standard of the product, and the use of the name by the USDA implies no approval of the product to the exclusion of others that may also be suitable.

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(peroxide value) and secondary oxidation products (oxidized fatty acids) were significantly lower in the oil containing 0.05% of the antioxidant than in the control. The citric acid-based antioxidant they evaluated, EPT-OILShield™, is a preparation of citric acid with potassium sulfate and aluminum silicate as adjuvants. It is formulated using a novel technology, code-named EPT for Energy Potential Technology, which was developed on the fundamental understanding that oxidation is also a function of molecular charge electronics (Park 2007). EPT works by modulating active energy charges and the electron transport chain within target substances to boost citric acid's natural antioxidant properties, while preserving its chemical characteristics. The EPT-enhanced citric acid based product is prepared using a proprietary method in which no thermal or chemical treatments are used.

In addition to the restaurant-type frying applications conducted by Cerretani and others (2008), food manufacturers are interested in alternatives to increase fry life of oils used for fried snack foods and to enhance the shelf life of the stored food. To help inhibit oxidation in stored fried food, there is a need for carry-through of an antioxidant to the food. Several researchers have reported that antioxidants added to frying oils carry over into the fried food to help inhibit oxidation during shelf-life storage (Fritsch and others 1975; Huang and others 1981; Nasirullah and Rangaswamy 2005; Warner and Laszlo 2005). This study expands the one by Cerretani and others (2008), which focused only on the effects of EPT-OilShield on the restaurant-style frying, for example, French fried potatoes. To determine the effects of this additive in frying snack foods, which is another common frying application, the objectives of this study were to determine the shelf life of tortilla chips fried in SBO with 2 levels of EPT-OILShield (0.05% and 0.5%) and a control and to determine the frying stability of the treated and untreated oils.

Materials and Methods

Materials

Commercially processed soybean oil (SBO) (ConAgra Foods, Omaha, Nebr., U.S.A.) with no additives (except citric acid added during processing) was purchased at a local grocery store. Tocopherol standards, α-, β-, γ-, δ-tocopherols, at 95% purity, were purchased from Matreya, Inc. (Pleasant Gap, Pa., U.S.A.). The citric acid-based antioxidant, Energy Potential Technology (EPT)-OILShield, was obtained from Elvisem AG (Rotkreuz, Switzerland). All chemicals and solvents were obtained from Sigma-Aldrich (St. Louis, Mo., U.S.A.), unless otherwise stated, and were ACS grade or better. Raw, yellow corn tortillas were obtained from a local grocery store.

Frying protocol

Frying protocol included intermittent batch frying of tortilla wedges at 180 °C with total heating/frying time of 65 h over a 6-d period. Each tortilla (15 cm dia) was cut into 6 equal wedges. Each 50 g batch of tortilla chips was fried for 90 s at 180 °C ± 2 °C in a 2 L capacity fryer (Natl. Presto Industries, Eau Claire, Wis., U.S.A.) containing 1200 g oil initially. Tortilla chips were fried every 30 min for 11 h each day for 6 d, except at every 5 h chip collection time, when 4 to 50 g batches of tortilla chips were fried to provide enough samples for analyses. Oil and chip samples were collected every 5 h until 65 h of frying. Fresh make-up oil (60 g) was added every 5 h after the oil and chip samples were collected. The frying experiment was duplicated. Tortilla chips were placed in 1 L wide-mouth glass jars with air in the headspace and the jars were closed with screw lids. Chips were aged in the jars in the dark for 0, 1, 2, and 4 mo at 25 °C, and then frozen at 0 °C until later analyses.

Instrumental and chemical analyses of oils

Fatty acid composition of the initial oil was determined by capillary gas chromatographic (GC) analysis with a Hewlett-Packard 5890 GC (Wilmington, Del., U.S.A.) equipped with a SP2330 column (30 m, 0.20 mm i.d., 0.20 micron film thickness) (Supelco, Bellefonte, Pa., U.S.A.). Column temperature was held at 190 °C for 5 min and temperature was programmed to 230 °C at 20 °C/min. Other GC conditions were: injector, 250 °C; detector, 260 °C. Analysis of α-, β, γ-, and δ- tocopherols by normal-phase HPLC was conducted in triplicate on a Varian ProStar (Varian Associates, Inc., Walnut Creek, Calif., U.S.A.) with a model 363 fluorescence detector. The detector was set at 290 nm for excitation and 330 nm for emission. The HPLC was fitted with a 5-μm Varian Inertsil Si column (250 × 4.6 i.d.). The isocratic solvent system, 0.5% 2-propanol in hexane, was pumped at 0.5 mL/min. Quantification of the tocopherols was made using external standard calibration. Initial oxidation of the fresh oils was measured in duplicate by peroxide value (AOCS method Cd 8-53) (AOCS 1998).

Total polar compound (TPC) levels of the fresh and used frying oils were determined in duplicate by the AOCS column chromatography method Cd 20-91(AOCS 1998). Free fatty acid (FFA) values were measured as percent oleic acid by AOCS method Ca 5a-40 (AOCS 1998).

Volatile compound analyses of tortilla chips

Hexanal content of the fresh and aged tortilla chips was analyzed in triplicate with a purge and trap apparatus equipped with a test tube adapter (Tekmar model 3000, Tekmar-Dohrmann Co., Cincinnati, Ohio, U.S.A.) coupled with a Varian model 3400 gas chromatograph (GC) and a Saturn model 3 ion trap mass spectrometer (MS) (Varian, Inc., Walnut Creek, Calif., U.S.A.). A 50 mg tortilla chip sample was placed in a 1.9 × 7.6 cm test tube and heated at 100 °C for 9 min preheat time. Volatile compounds were trapped on a 30.5 cm Tenax nr 1 trap, with 10 min sample purge time, 170 °C for 6 min desorbing, 180 °C MCS desorb temperature, 160 °C GC transfer line and valve temperature. Volatile compounds were introduced onto a DB-1701 GC capillary column (30 m × 0.32 mm with 1 micron film thickness) (J & W Scientific, Folsom, Calif., U.S.A.). The column was held at −20 °C for 2 min, and then heated from −20 to 233 °C at 3 °C/min. Column helium flow rate was 2 mL/min with 28 mL/min injector split vent flow. The GC injector was set at 240 °C and the line to the mass spectrometer was set at 230 °C. The ion trap MS operated in EI mode with a mass scan range of 23 to 400 m/z over 0.8 s. The filament emission current was 25 micro amps, axial modulation was 2.1 volts, manifold heater was set at 160 °C and filament/multiplier delay was 2.5 min. Compound structural identifications were made both from spectral comparisons with the NIST 92 mass spectrometry library (Varian, Inc.) and from retention time comparisons with standard compounds.

Sensory analysis of tortilla chips

A 15-member analytical, descriptive sensory panel, trained and experienced in evaluating fried foods were presented with 2 g crushed tortilla chip samples in 59.2 mL (2 oz) plastic soufflé cups with snap-on lids (Solo Cup Co., Urbana, Ill., U.S.A.). All sensory evaluations were conducted at our research center in a panel room with individual booths, temperature control and with red lighting to mask color differences between samples (Warner 1995). Panelists rated the tortilla chips for rancid flavor intensity on a 10-point intensity scale with 0 = no intensity and 10 = strong intensity (Warner and Gupta 2005). Each panelist received 4 coded, randomized samples of each fry time (for example, 25 h) at each storage time (for example, 2 mo) per session.

Statistical analysis

Data were evaluated by analysis of variance (ANOVA) and Duncan's multiple range test when *F* values were significant (Snedecor 1956). ANOVA was conducted using Microcal Origin 6.0 (Microcal Software, Inc., Northampton, Mass., U.S.A.). Statistical significance was expressed at the $P \leq 0.05$ level unless otherwise indicated.

Results and Discussion

EPT-OILShield is a natural, oil and water-soluble antioxidant with citric acid as its main active ingredient. Adjuvants in the composition include aluminum silicate and potassium sulfate. All EPT-OILShield ingredients are classified as generally recognized as safe (GRAS) by the U.S. Food and Drug Administration (FDA). The fine powder was dispersed in the SBO samples at 0.5% and 0.05% with a stir bar for 5 min. The starting SBO had a PV of 0.3 meq/kg, indicating good initial quality. Fatty acid composition of the initial soybean oil was 10.7% C16:0, 4.9% C18:0, 24.4% C18:1, 53.1% C18:2, and 7.2% C18:3.

Frying stability

Free fatty acids (FFA) and total polar compounds (TPC) formed during frying are indicators of deterioration levels in the oil. FFA analysis was used to determine effects of hydrolysis in the frying oils. FFA increased with increasing frying time in all oils (Figure 1); however, the SBO with 0.05% EPT-OILshield had significantly lower amounts of FFA formed than in the control at all sampling times after 5 h of frying. On the other hand, the use of the higher dose of 0.5% EPT-OILShield showed increased FFA formation at all sampling times compared to the control and to the oil with only 0.05% of the additive. The development of TPC followed a similar trend as for FFA. TPC increased with increasing frying time in all oils (Figure 2). SBO with 0.05% EPT-OILShield had significantly lower amounts of TPC formed than in the control at sampling times from 5 to 65 h. SBO with the higher 0.5% of the antioxidant showed increased TPC formation at all sampling times compared to the control and to the oil with 0.05% EPT-OILShield. The addition of higher doses of EPT-OILShield can create a situation of increased polarization of the compounds, increasing the formation of FFA and TPC being detected. Higher dosage levels alter the isoelectric fields thus obtainingless desirableresults at higher dosages. Our results agree with those from frying studies with French fried potatoes by Cerretani and others (2008) who found that 0.05% EPT-OILShield was significantly better than a SBO control and a SBO with 0.1% EPT-OILShield for limiting peroxide value and oxidized fatty acid development.

Tocopherol retention in frying oil and fried food

Gordon and Kourimska (1995) reported that additives such as ascorbyl palmitate and rosemary extract retarded losses of natural tocopherols and that the stability of rapeseed oil decreased as the tocopherols were consumed. We report similar results in this study with EPT-OILShield. As the number of hours of frying increased, the levels of the tocopherols decreased, with alpha and delta tocopherol losing the least amounts (Table 1). After 65 h of frying, the control retained the least percent of alpha and gamma tocopherols (Table 1). The oil with the 0.05% EPT-OILShield retained the highest percent of the gamma and delta tocopherols, which may have helped limit the amount of deterioration in that frying oil (Figure 1 and 2) because they are considered potent antioxidants *in vitro* (Frankel 2005). For example, after 65 h of frying, 68% of gamma tocopherol was retained in the oil with 0.05% of the additive compared to only 33% retained in the control and

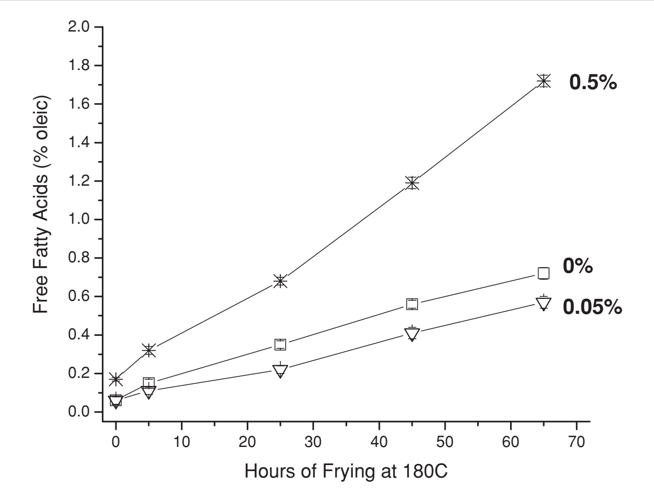

Figure 1 — Free fatty acids (% oleic) in soybean oil (SBO) with 0%, 0.05%, or 0.5% EPT-OILShield and used for frying tortilla chips at 180 °C.

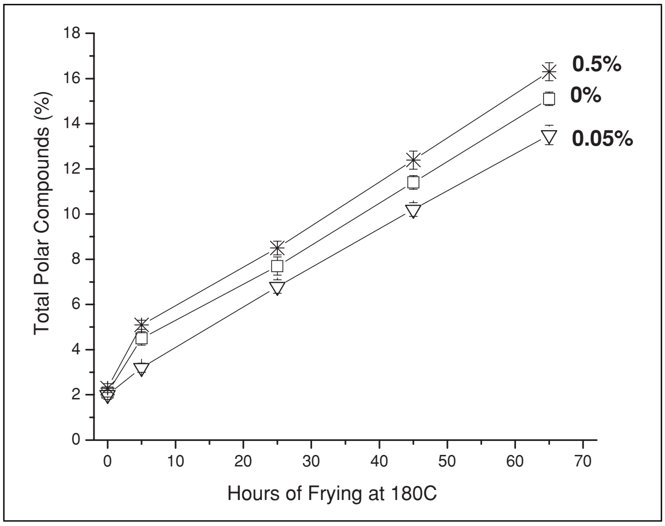

Figure 2 — Total polar compounds (%) in soybean oil (SBO) with 0%, 0.05%, or 0.5% EPT-OILShield and used for frying tortilla chips at 180 °C.

Table 1 — Tocopherol amounts[a] (ppm) retained in soybean oil with 0%, 0.05%, or 0.5% EPT-OILShield for up to 65 h of frying.

	Hours of frying	0%	0.05%	0.5%
Alpha	0	167a	165a	169a
	25	164a	171a	163a
	45	155a	166a	168a
	65	154a	158a	169a
α retention at 65 h (% of baseline)		93%	96%	100%
Gamma	0	717a	716a	701a
	25	608a	630b	579c
	45	499a	558b	520c
	65	233a	482b	438c
γ retention at 65 h (% of baseline)		33%	68%	62%
Delta	0	188a	185a	193a
	25	178a	184a	166a
	45	160a	174a	156a
	65	153a	162a	144a
δ retention at 65 h (% of baseline)		81%	88%	75%

[a]Values with no letter in common in each row are significantly different.

62% retained in the oil with 0.5% of the EPT-OILShield. Delta tocopherol retention was highest at 88% in the oil with 0.05% additive, followed by 81% retention in the control and 75% in the oil with 0.5% additive. Retention amounts were similar in the 3 oils for alpha tocopherol.

On the other hand, a different pattern of tocopherol retention was noted in the oils extracted from the tortilla chips (Table 2). Although tocopherols were lost as the amount of heating and frying time increased, more significant losses were noted in the aged chips during storage. Moreover, there was a significant difference between the tocopherol retention of the oil extracted from the chips with 0.05% EPT-OILShield and the control. For example, in chips fried in oils used for 65 h of frying, the chips fried in the control retained only 9% gamma tocopherol compared to 38% in the chips with 0.05% additive, and 8% in the chips with 0.5% additive. The pattern for delta tocopherol retention was similar to that of gamma tocopherol with the most retention noted in the chips with 0.05% additive at 77%, followed by 58% in both the samples with 0.5% antioxidant and in the control. The higher retention of gamma and delta tocopherols in the oil extracted from chips fried in the oil with 0.05% additive could help to inhibit oxidation in the tortilla chips during storage.

Table 2—Tocopherol amounts[a] (ppm) retained in tortilla chips fried in soybean oil with 0%, 0.05%, or 0.5% EPT-OILShield for up to 65 h of frying.

	Hours of frying	Months of chip storage	0%	0.05%	0.5%
Alpha	0	0	167a	165a	169a
	25	0	145a	129a	139a
	25	2	43a	121b	89c
	25	4	21a	78b	45c
	45	0	149a	110b	130ab
	45	2	8a	105b	108b
	45	4	0a	44b	25b
	65	0	75a	98b	74a
	65	2	22a	85b	64b
	65	4	13a	44b	33ab
α retention at 65 h/4 m (% of baseline)			8%	27%	20%
Gamma	0	0	717a	716a	701a
	25	0	381a	572b	306c
	25	2	325a	568b	251c
	25	4	216a	440b	149c
	45	0	342a	478a	292c
	45	2	189a	462b	190a
	45	4	126a	387b	143a
	65	0	335a	406b	242c
	65	2	215a	269b	174c
	65	4	61a	267b	55a
γ retention at 65 h/4 m (% of baseline)			9%	38%	8%
Delta	0	0	188a	185a	193a
	25	0	159ab	174a	144b
	25	2	152ab	170a	140b
	25	4	146a	159a	115b
	45	0	155a	167a	139a
	45	2	123a	164b	137a
	45	4	78a	145b	120b
	65	0	146a	144a	133a
	65	2	138a	147a	135a
	65	4	109a	142b	114a
δ retention at 65 h/4 m (% of baseline)			58%	77%	58%

[a]Values with no letter in common in each row are significantly different.

Oxidative stability of tortilla chips

Hexanal was used to monitor the oxidation of the aged tortilla chips after ambient temperature storage because it is a major volatile compound formed during oxidation of linoleic acid (Frankel 2005), which is the predominant fatty acid in this SBO sample. Hexanal analysis was conducted on tortilla chips aged after 2 and 4 mo at 25 °C except for the samples fried in the 65 h oil, which were also tested after 1 mo of aging. Frying tortilla chips does not deteriorate oils as rapidly as some other types of food that contain more water such as potato chips. Therefore, these oils did not have much deterioration until approximately 25 h of frying (Figure 1 and 2), so testing of chip samples was not conducted on samples fried in oils used less than 25 h.

In the unaged tortilla chips fried in oils used for 25 h (Figure 3A), hexanal levels were similar between oil types and ranged from 1 to 2 ppm; however, after 2 mo storage, significant differences were detected. For example, chips fried in oil with 0.05% EPT-OILShield had the lowest level of hexanal (2 ppm), followed by the sample with 0.5% EPT-OILShield with 4 ppm and the control with 7.5 ppm hexanal. At the 4 mo storage time, the chips fried in the SBO control had significantly higher hexanal content at 18 ppm. Tortilla chips fried in SBO with 0.05% EPT-OILShield were the most stable after 4 mo of aging with only 3 ppm hexanal formed.

In chips fried in oils used for 45 h (Figure 3B), a similar pattern of hexanal formation was observed as in the chips fried in oils used for 25 h (Figure 3A). For example, the chips fried in the SBO control had significantly more hexanal formed after 2 mo storage than chips fried in the treated oils. By 4 mo of storage, hexanal was significantly lower in the chips fried in oil with 0.05% EPT-OILShield and the next lowest amount was in chips fried in the oil with 0.5% of the antioxidant. The chips fried in SBO control had a significantly higher amount of hexanal with 24 ppm.

As the frying oil deterioration increases over time, the foods fried in the more abused oil are more susceptible to oxidation during storage (Frankel 2005; Warner and Gupta 2005). Therefore, the oils used for 65 h of frying produced the least stable fried food with the chips fried in the control having the highest amounts of hexanal at 1, 2, and 4 mo storage (Figure 3C). Tortilla chips fried in SBO with 0.05% EPT-OILShield were the most stable after all storage times compared to chips fried in the other 2 oils as indicated by significantly less hexanal formation. The addition of 0.05% or 0.5% EPT-OILShield significantly improved the oxidative stability of chips compared to those fried in untreated SBO; however, the 0.05% level was the most effective in limiting formation of hexanal.

Fried food stability

Sensory analysis of rancid flavor intensity was conducted on tortilla chips sampled after the chips were aged for 0, 2, and 4 mo after 25, 45, and 65 h of intermittent frying (Figure 4). The 10-point scale used represents a range of intensity of 0 = no rancid flavor to 10 = strong rancid intensity. The portion of the scale from 0 to 3 represents weak rancid flavor intensity range; 3 to 6 represents moderate rancid flavor range; and 6 to 10 represent strong rancid flavor intensity. Panelists were presented with reference standards of oxidized tortilla chips representative of none, weak, moderate, and strong rancid flavor intensity before evaluation of the chips in this study began. In most polyunsaturated oils such as SBO, with high amounts of linoleic acid, rancid flavor intensity increases as the length of storage time increases (Warner and Gupta 2005). In this study, as expected, rancid flavor intensity increased faster with increasing storage time for chips fried in untreated SBO than for the treated oils. At the 25 h frying time, the addition of the EPT-OILShield was effective in inhibiting that off-flavor to less than 0.5 intensity in chips fried in oil with 0.05% of the additive and to a rancid intensity of less than 1 for the chips fried in SBO with 0.5% additive after 4 mo of storage (Figure 4A). However, the chips fried in the control had a rancid intensity of 2 after 2 mo storage and a moderate intensity of 4.1 after 4 mo. The same pattern was repeated for the chips fried in oil used for 45 h for the control and the oil with 0.05% additive; however, the chips fried in the oil with 0.5% antioxidant had a rancid flavor intensity of 2 by 4 mo. For the chips fried in oils used for 65 h, the chips fried in the control were moderately rancid by 4 mo, but the chips fried in oil with 0.05% EPT-OILShield had a rancid flavor intensity of only 0.2 after 2 mo of storage, indicating good oxidative stability.

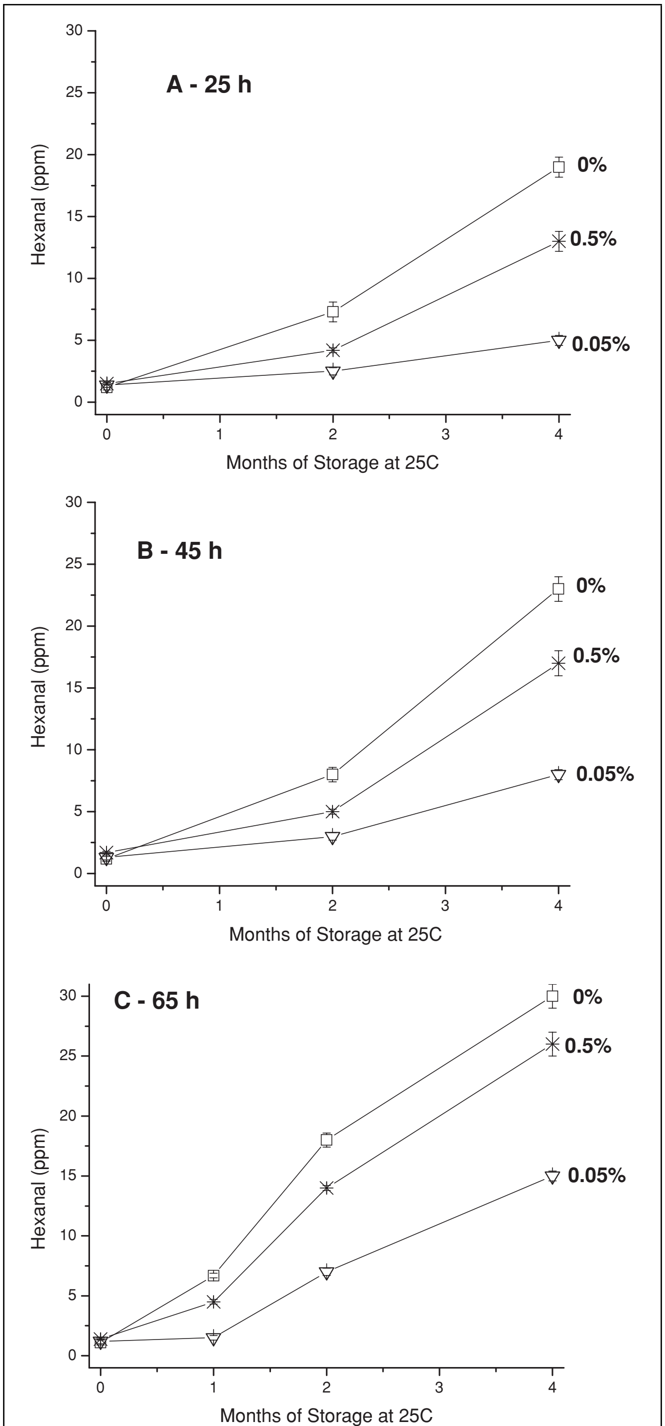

Figure 3—Hexanal in tortilla chips fried in soybean oil (SBO) with 0%, 0.05%, or 0.5% EPT-OILShield for 25, 45, or 65 h at 180 °C and aged for up to 4 mo at 25 °C.

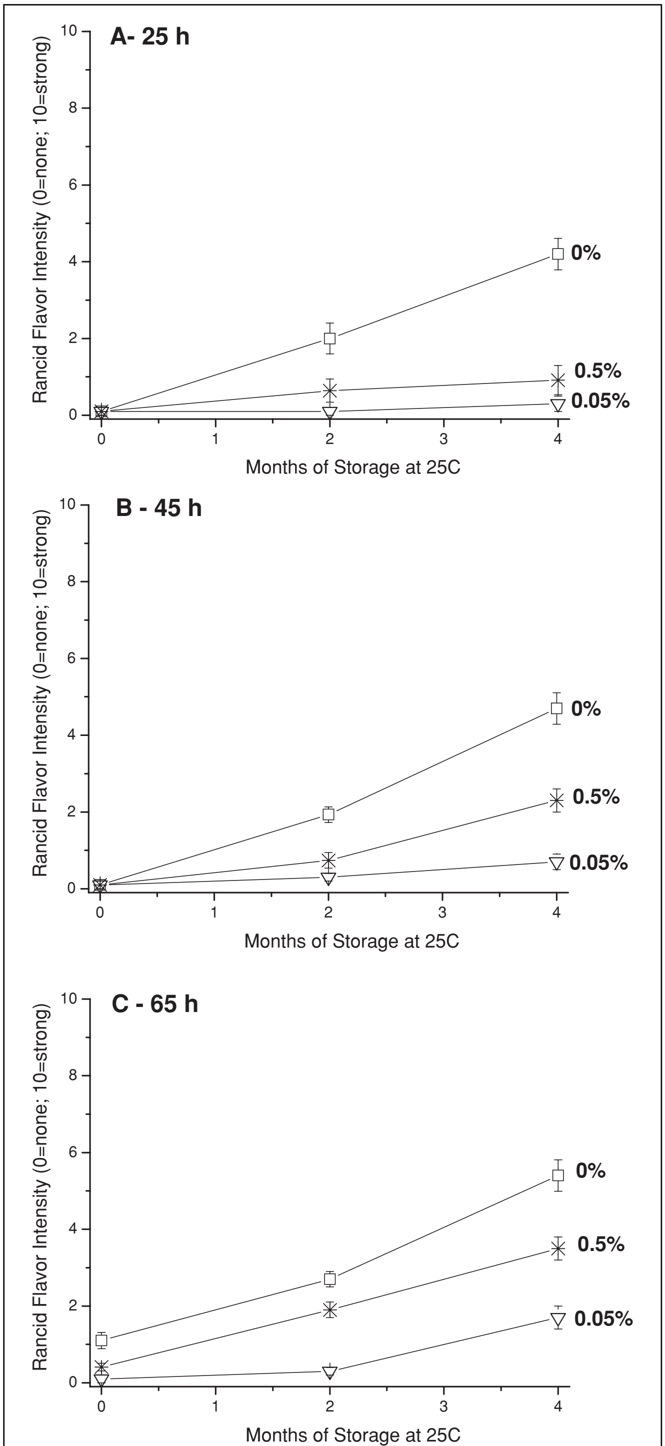

Figure 4—Rancid flavor intensity of tortilla chips fried in soybean oil (SBO) with 0%, 0.05%, or 0.5% EPT-OILShield for 25, 45, or 65 h at 180 °C and aged for up to 4 mo at 25 °C.

Conclusions

This study showed that SBO could have improved fry life as measured by FFA and TPC with the addition of 0.05% of the citric acid-based antioxidant. The effect of the antioxidant added to the oil carried over into the fried food. In tortilla chips fried in oils used for 25, 45, or 65 h, the rancid flavor intensity was significantly lower in chips fried in oil with 0.05% EPT-OILShield than in the control or the oil with 0.5% additive.

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